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(57) Abstract

A process for the production of a wood composite (such as fiberboard) by a wet-felted process having improved mechanical and physical properties and surface characteristics includes the use of a binder comprising an organic isocyanate, such as 4,4-diphenylmethane-diisocyanate ("MDI").

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ORGANIC ISOCYANATES AS BINDERS FOR WOOD COMPOSITES

BACKGROUND OF THE INVENTION

Field of the Invention

The invention relates generally to the production of a wood composite having superior surface and overall quality, and more particularly to the use of an organic isocyanate binder to manufacture a pressed and/or molded wood composite with superior surface quality.

Description of Related Technology

Wood composites, e.g., hardboard or fiberboard, may be formed in desired shapes and sizes depending on the intended use; examples include doorfacing or doorskin which is applied to a door body, and so-called smooth-two-sided ("S2S") hardboard for store fixtures and other uses. The principal processes for the manufacture of wood composites include (a) wet felted/wet pressed or "wet" processes, (b) dry felted/dry pressed or "dry" processes, and (c) wet felted/dry pressed or "wet-dry" processes.

Generally in a wet process, cellulosic fillers or fibers (e.g., woody material which is subjected to fiberization to form wood fibers) are blended in a vessel with large amounts of water to form a slurry. The slurry preferably has sufficient water content to suspend a majority of the wood fibers and preferably has a water content of at least 90 weight percent, at most preferably at least 96-98.5 weight percent. The slurry is deposited along with a synthetic resin binder, such as a phenol-formaldehyde resin, onto a water-pervious

support member, such as a fine screen or a Fourdrinier wire, where much of the water is removed to leave a wet mat of cellulosic material having, for example, a moisture content of about fifty weight percent. The wet mat is transferred from the pervious support member to a press and consolidated under heat and pressure to form the molded wood composite.

In a dry process, the cellulosic fibers are generally conveyed in a gaseous stream (or by mechanical means) rather than a liquid stream. For example, the cellulosic fibers may be first coated with a thermosetting resin binder, such as a phenol-formaldehyde resin. The fibers are then randomly formed into a mat by air blowing the resin-coated fibers onto a support member. The mat, typically having a moisture content of less than 30 wt.% and preferably less than 10 wt.%, is then pressed under heat and pressure to cure the thermosetting resin and to compress the mat into an integral consolidated structure.

A wet-dry forming process may also be used to produce wood composites. Generally, in a wet-dry process, a slurry (described above) is formed of water, a cellulosic fiber, and a resin binder. Sufficient water is then drained by vacuum and press rolls from the slurry to form a wet mat. Further water is then removed from the wet mat by evaporation, which is preferably facilitated by the application of heat, in order to form a dried mat. The dried mat is then pressed under heat to form the wood composite.

Preferably, a wet-dry process begins by blending cellulosic or wood fiber raw material in a vessel with large amounts of water having a pH of

less than 7 to form a slurry. This slurry is then blended with the resin binder. The blend is then deposited onto a water-pervious support member, where a large percentage (e.g., 50 percent or more) of the water is removed, thereby leaving a wet mat of cellulosic material having a water content of about 40 wt.% to about 60 wt.%, for example. Further water may be removed in a second step, in which case these two steps may be referred to respectively as the primary water removal step and the secondary water removal step. This wet mat is then transferred to an evaporation zone where much of the remaining water is removed by evaporation, for example by heating the wet mat. The mat may be further dried in a second evaporation step, in which case these two evaporation steps may be referred to respectively as the primary evaporation step and the secondary evaporation step. (These steps are commonly referred to as "drying" steps.) The dried mat preferably has a moisture content of less than about 10 wt.%. The dried mat is then transferred to a press and consolidated under heat and pressure to form the wood composite which may be, for example, a flat board or any other desired shape depending on the intended use of the product.

Wood composites produced according to the processes described above may, however, have poor surface quality. Poor surface quality is indicated by a wood composite having a porous or open surface, inadequate consolidation along the edges or corners of the wood composite and/or poor definition of wood grain which is often embossed on the surface of the wood composite. Poor surface quality is also indicated where the wood composite exhibits poor internal bond and strength, especially at the edges

of the wood composite. For example, poor surface quality is shown where there appears to be layers within the composite which are "flaky" and can be easily peeled away; this phenomenon may be referred to as a lack of surface tightness. Where there is poor surface quality, the wood composite may easily break apart and has limited useful life, thereby making the product unsatisfactory.

The above-described effects of a poorly bonded surface often result from undesirable pre-curing of the thermosetting phenol-formaldehyde resin prior to full curing of the resin in its final shape in the press. For example, this pre-curing can occur (a) during drying of the wood fiber mat and/or (b) in the press before the final thickness of the product is achieved.

In order to help prevent the problems associated with poor surface quality, various procedures have been attempted. For example, a urea solution has been used as a surface treatment. The urea converts to ammonia under heat, which then plasticizes the fibers during consolidation. However, the use of urea has several disadvantages, including the relatively high application rate which is required (up to two grams of urea solids per square foot), contribution to the build up of undesirable material (e.g., a film which includes carbon and other materials, commonly referred to as a "carbon film") on the die surfaces, promotion of corrosion on unplated die or platen surfaces, and the presence of ammonia in the press exhaust stream. The carbon film may damage the final product and/or the die surfaces and is difficult to remove from the die surfaces. The presence of ammonia in the exhaust gases is of particular concern if thermal

oxidation is used as a pollution control measure, because the ammonia can convert to oxides of nitrogen (NO_x), which are hazardous. It is therefore desirable to reduce or eliminate the need for a urea treatment during pressing of the wood product.

Also, when drying oils (for example tung oil, linseed oil, or palm oil) are used as binders, it typically is necessary to "bake" the wood product after pressing in order to achieve satisfactory mechanical properties. When a wood product is baked, it typically is heated to about 250°F - 300°F for about thirty to sixty minutes. However, baking is undesirable due to various factors, including fire hazard, air pollution, and additional energy and labor costs associated with this procedure.

The surface of the wood composite may also be improved by postpress tempering with drying oils. Many different types of tempering oils are conventionally used, including linseed oil, soybean oil, tung oil, oiticica oil, and unsaturated fatty acid esters. However, the use of these oils increases both cost and production time. In addition, use of such oils may be environmentally undesirable. It is therefore desirable to reduce or eliminate the need for such postpress treatment of the molded wood product.

SUMMARY OF THE INVENTION

It is an object of the invention to overcome one or more of the problems described above.

Accordingly, the invention provides a process for producing a wood composite which has superior surface quality and strong internal bonding.

According to the invention, an organic isocyanate resin is used as a binder in a wet-dry hardboard process to produce a wood composite. The organic isocyanate is preferably used to replace conventional binders such as phenolic resins.

In one embodiment of the invention, the organic isocyanate binder is combined with cellulosic material and process water having moderate acidity to form a slurry. The slurry is drained to form a wet mat, followed by drying to form a dried mat, preferably having a reduced moisture content. The dried mat is then consolidated in a hot press.

The inventive process preferably obviates the need for further treatment, e.g., tempering with oils, to provide a wood composite with high surface quality.

Other objects and advantages of the invention will be apparent to those skilled in the art from the following detailed description, taken in conjunction with the appended claims.

DETAILED DESCRIPTION OF A PREFERRED EMBODIMENT

According to the invention, a binder is provided in combination with a process which will preferably improve the surface characteristics and internal bond strengths of wood composites. The binder is blended with a cellulosic filler, e.g., wood fiber, and formed into a wood composite.

The binder provided by the invention is an organic isocyanate, which is utilized to replace conventional hardboard binders in the production of wood composites. The isocyanate binder replaces such conventional hardboard binders as phenolic resins or tung oil. The isocyanate binder is

blended with a cellulosic filler (e.g., wood particles or fibers) and formed into a wood composite. Such a procedure overcomes the problems described above, e.g., precuring of the resin, yet allows for strong internal bonding of the wood composite. For example, a wood composite produced according to the inventive method may have an excellent internal bond strength (which may be the result of branching and cross-linking of the binder) superior to the bond strength resulting from the use of other resins which do not cross-link upon curing.

Preferably, 4,4-diphenylmethane-diisocyanate ("MDI") is the isocyanate utilized in accordance with the inventive process. MDI may be any commercial grade such as those sold under the trade designations Miles Mondur 451 (available from Miles Laboratories of Elkhart, Indiana) and ICI MF-184 (available from ICI United States, Inc. of Wilmington, Delaware), which are mixtures of MDI monomers and corresponding oligomers. Emulsifiable MDI, such as those designated Miles XW-126 and ICI Rubinate MF-178 can also be used in accordance with the invention. Emulsifiable MDI is optionally pre-emulsified with water prior to addition into the fiber slurry.

Numerous other organic isocyanates are suitable for use with the invention, and include any monomeric or polymeric isocyanates that have at least two reactive isocyante (-NCO) groups. Specific examples are: toluene-2,4-diisocyanate, toluene-2,6-diisocyanate, triphenylmethane triisocyanate, dimethyl diphenylmethane-4,4-diisocyanate. Further examples are disclosed in U.S. Patent No. 4,209,433 to Hse, the disclosure of which is incorporated herein by reference.

The inventive process may have a beneficial effect on board consolidation in the hot press. The outer layers of board produced by the inventive process preferably are better consolidated than board made with (a) conventional (e.g., phenolic) resins or (b) processes which use a urea-based surface treatment. As a result, because the inventive process typically provides excellent surface quality, the need for further treatment, generally necessary to improve the surface quality of the wood composite, may be reduced or obviated. For example, the quantity of post-press applied tempering oil required to bind the outer layers of molded door facings may be significantly reduced or eliminated. Further, the need for urea treatment, described above, may also be eliminated. Because the need for further treatment, e.g., tempering oils and urea, may be obviated, there may be a reduction or elimination of the utilization and production of undesirable materials. For example, ammonia and oxides of nitrogen may be eliminated from the press exhaust.

Further, the inventive process also preferably eliminates the need for post-press "baking" which is typically required in order to achieve satisfactory mechanical properties when using drying oils as binders. When the inventive process is utilized in place of processes using drying oils, non-baked boards may have better mechanical properties than boards made with drying oils. As a result, the post-press baking procedure is preferably eliminated.

An example of the inventive process is described below in conjunction with the manufacture of a fiberboard by a wet-dry process. The resulting

fiberboard may be in the form of a doorfacing or doorskin which is then applied to a door body to make a final product in the form of a door. The final product can also be a fiberboard in the form of smooth-two-sided ("S2S") hardboard suitable for various industrial and commercial applications.

According to the process, process water is preferably first incorporated with cellulosic particles or fillers, e.g., wood which has been fiberized and prepared according to any known method to form wood fibers. This slurry preferably has a temperature of about 60°F to about 200°F (about 15°C to about 93°C) and a pH of about 3.5 to about 5.5.

The slurry of process water and cellulosic fibers is then combined with a binder comprising an organic isocyanate. Preferably, 4,4-diphenylmethane-diisocyanate ("MDI") is selected as the binder resin, as described above. Other organic isocyanates, or their equivalents, may be used in place of MDI, and it may be desirable to utilize a combination of two or more different organic isocyanates. The MDI content of this slurry, based on dry fiber weight, is preferably about 0.25 wt.% to about 2 wt.%. The order in which these ingredients are mixed to form the slurry is not essential; the ingredients may be incorporated in any order, for example by first combining the process water and binder resin, followed by adding the wood fibers. The slurry which is formed is preferably mixed or stirred to sufficiently combine the ingredients.

Although the binder may consist essentially of an organic isocyanate, additional binders may optionally be utilized. Where such a combination is desired, the slurry formed of the

organic isocyanate binder, process water and cellulosic material may be combined with an additional binder resin. Such additional resin is preferably a phenolic resin, such as a phenol-formaldehyde resin or an o-cresol-formaldehyde resin. It is generally not desirable to combine the organic isocyanate resin with the additional, phenolic resin prior to formation of the slurry. Rather, it is preferable to form a slurry of an organic isocyanate binder, process water and cellulosic material and then blend the slurry with such an additional, phenolic resin.

Further, the isocyanate binder resin may optionally be combined with a drying oil (e.g., linseed oil or tung oil) prior to addition to the fiber slurry. As opposed to the additional, phenol-formaldehyde resin or an o-cresol-formaldehyde resin (described in the preceding paragraph) the drying oil may be beneficially combined with the organic isocyanate binder resin prior to addition to the fiber slurry.

The slurry or mixture is placed on a water-pervious screen which removes excess water from the slurry by draining, thereby forming a wet mat. After this primary water removal step, the mat is optionally subjected to a secondary water removal step, which may use mechanical means to remove further water. After the water removal step(s), the wet mat preferably has a moisture content of less than about 70 wt.%, more preferably about 40 to about 60 wt.%, and most preferably about 50 wt.%.

The wet mat is preferably next transported to a drying zone, where the moisture content is still further reduced, as by heating. This drying is optionally performed in two steps, as stated

above. For example, the mat may be dried at a temperature in the range of about 250°F to about 450°F (about 120°C to about 232°C) for about 1 to about 6 hours. As the mat leaves the drying zone as a dried mat, the moisture content is preferably less than about 10 weight percent, e.g., about 0 to about 10 weight percent. The most preferred moisture content is near zero (e.g., between 0.1 and 2 weight percent).

Subsequent to drying, the dried mat is placed in a hot press where the mat is consolidated under heat and pressure to produce a wood composite. The pressing temperature is variable depending upon the materials and other process parameters selected. However, the pressing temperature is preferably greater than about 400°F (about 200°C), and most preferably in the range of about 480°F to about 500°F (about 250°C to about 260°C). Desirable pressing times may also vary depending upon the materials and other process parameters selected, however, the dried mat is preferably pressed for about 60 seconds to about 150 seconds for a product that ranges from 1/8-inch to 1/4- inch thick. More specifically, for 1/8-inch thick hardboard, the pressing time is preferably about 60 seconds, and for a 0.22-inch thick product, the pressing time is preferably about 105 seconds.

After the wood composite has been pressed, it may be immediately transported for coating, gluing, staining, or other finishing to complete a desired product for commercial use. Because the wood composite preferably has superior surface quality, including complete consolidation of the mat along its edges and corners, without any further treatment, the need for tempering the product with

oils is preferably either greatly reduced or eliminated. Further, the inventive process preferably does not require the use of pre-press sealers generally utilized to improve the surface quality of the wood composite.

The inventive process also preferably prevents or significantly reduces the generation of oxides of nitrogen (NO_x) when thermal oxidation is used as a pollution control measure for press emissions, a disadvantage generally encountered when using urea-based treatments, as described above.

A comparison was made between boards made according to the inventive process and boards using tung oil as a binder. The boards made according to the inventive process were made by the method described above, with the following particular parameters: (a) the above-described slurry was formed to at a moisture content of 99 wt.% (i.e., a 1% "consistency" in the slurry); (b) a press temperature of about 480°F; and (c) a press time of about 105 seconds. The emulsifiable MDI disclosed above (ICI Rubinate MF-178) was selected as the binder in the inventive process. As shown in the Table below, MDI content levels of 0.75 wt.% and 1 wt.% (based on the weight of cellulosic filler) were used. The products were smooth-two-sided ("S2S") flat hardboard pressed at thicknesses of 0.22 inch and 0.19 inch, respectively. The control boards used 0.7 wt.% tung oil and were pressed at a thickness of 0.22 inch.

The table below outlines the results of the comparative example. As can be seen, the average internal bond strength (IB) and modulus of rupture (MOR) of the boards made by the inventive process were significantly higher than those values

for the control boards made with tung oil. Both tempered and non-tempered hardboards made by the inventive process passed the perforation test and the cleavage test (values greater than 60 lbs.), thereby indicating good surface quality. Further, cleaning of the pressing equipment was facilitated by use of the inventive process, and use of a cleaning solvent was unnecessary with the equipment used in the inventive process.

Binder	Thickness	Baking	Tempering	Specific Gravity	IB (psi)	MOR (psi)	Cleavage (lbs)
Control:							
0.7% tung oil	0.22"	Yes	No	0.91	127	6234	87
0.7% tung oil	0.22"	Yes	Yes	0.90	107	6159	88
Trial:							
0.75% MDI	0.22"	No	No	0.90	164	6255	79
0.75% MDI	0.22"	No	Yes	0.90	177	6481	96
1% MDI	0.19"	No	No	1.06	328	8863	90
1% MDI	0.19"	No	Yes	1.06	338	9323	107

The foregoing detailed description is given for clearness of understanding only, and no unnecessary limitations should be understood therefrom, as modifications within the scope of the invention will be apparent to those skilled in the art.

CLAIMS

1. A process for the preparation of a wood composite, comprising:

(a) forming a slurry comprising water, a cellulosic filler, and a binder comprising an organic isocyanate resin;

(b) draining sufficient water from the product of step (a) to form a wet mat;

(c) removing water from said wet mat by drying to form a dried mat; and

(d) pressing said dried mat under heat to form a wood composite.

2. The process of claim 1 wherein said organic isocyanate resin comprises 4,4-diphenylmethane-diisocyanate.

3. The process of claim 1 wherein said dried mat is pressed at at least about 400°F.

4. The process of claim 3 wherein:

said mat is pressed at about 430°F to about 500°F.

5. The process of claim 1 wherein:

the weight of said organic isocyanate resin is about 0.25% to about 2% of the weight of said cellulosic filler.

6. The process of claim 1 wherein:

said slurry is formed at a temperature of about 200°F or less.

7. The process of claim 6 wherein:

said slurry is formed at a temperature of about 60°F to about 200°F.

8. The process of claim 1 wherein:

said process water has a pH of about 3.5 to about 5.5.

9. The process of claim 1 wherein:

said wet mat has a moisture content of less than about 70 wt.%.

10. The process of claim 1 wherein:

said dried mat has a moisture content of up to about 10 wt.%.

11. The process of claim 1 wherein:

said binder further comprises a drying oil.

12. The process of claim 1, further comprising:

adding a phenolic resin to said slurry prior to step (b).

13. The process of claim 1 wherein:

said binder consists essentially of an organic isocyanate.

14. The process of claim 1 wherein:

said resin is selected from the group of toluene-2,4-diisocyanate, toluene-2,6-diisocyanate, triphenylmethane triisocyanate, and dimethyl diphenylmethane-4,4-diisocyanate.

15. The process of claim 1 wherein:

said cellulosic filler comprises wood fiber.

16. The process of claim 15 wherein:

said product of step (a) has a moisture content of about 99 wt.%.

17. A process for the preparation of a wood composite, comprising:

(a) forming a slurry comprising water, a cellulosic filler, and a resin comprising 4,4-diphenylmethane-diisocyanate, said slurry having a moisture content of at least about 90 wt.%;

(b) draining sufficient water from the product of step (a) to form a wet mat having a moisture content of less than about 70 wt.%;

(c) removing water from said wet mat by drying to form a dried mat having a moisture content of less than about 10 wt.%; and

(d) pressing said dried mat at a temperature greater than about 400°F to form a molded wood composite.

18. A method for resisting pre-cure in a process for the preparation of a wood composite by a wet-dry forming process wherein a cellulosic mat comprising a binder is formed, comprising:

selecting a binder comprising an organic isocyanate.

19. A method for improving the surface quality of a molded wood composite prepared by a wet-dry forming process wherein a cellulosic mat comprising a binder is formed, comprising:

selecting a binder comprising an organic polyisocyanate for use in said wet-dry forming process.

20. A process for the preparation of a wood composite, comprising:

(a) forming a slurry comprising water, a cellulosic filler, and a binder comprising an organic isocyanate resin;

(b) forming a mat from the product of step (a); and

(c) pressing said mat under heat to form a wood composite.

21. The process of claim 20 wherein:

said organic isocyanate resin comprises 4,4-diphenylmethane-diisocyanate.

22. The process of claim 20 wherein:

said dried mat is pressed at at least about 400°F.

23. The process of claim 20 wherein:

said mat is pressed at about 430°F to about 500°F.

24. The process of claim 20 wherein:

the weight of said organic isocyanate resin is about 0.25% to about 2% of the weight of said cellulosic filler.

25. The process of claim 20 wherein:

said slurry is formed at a temperature of about 200°F or less.

26. The process of claim 20 wherein:
said mat has a moisture content of at least about 90 wt.%.

27. The process of claim 20 wherein:
said cellulosic filler comprises wood fiber.

AMENDED CLAIMS

[received by the International Bureau on 20 January 1995 (20.01.95); original claims 3,4,18,19,22 and 23 amended; remaining claims unchanged (5 pages)]

1. A process for the preparation of a wood composite, comprising:

(a) forming a slurry comprising water, a cellulosic filler, and a binder comprising an organic isocyanate resin;

(b) draining sufficient water from the product of step (a) to form a wet mat;

(c) removing water from said wet mat by drying to form a dried mat; and

(d) pressing said dried mat under heat to form a wood composite.

2. The process of claim 1 wherein said organic isocyanate resin comprises 4,4-diphenylmethane-diisocyanate.

3. The process of claim 1 wherein said dried mat is pressed at a temperature of at least about 400°F.

4. The process of claim 3 wherein:

said dried mat is pressed at a temperature of about 430°F to about 500°F.

5. The process of claim 1 wherein:

the weight of said organic isocyanate resin is about 0.25% to about 2% of the weight of said cellulosic filler.

6. The process of claim 1 wherein:

said slurry is formed at a temperature of about 200°F or less.

7. The process of claim 6 wherein:

said slurry is formed at a temperature of about 60°F to about 200°F.

8. The process of claim 1 wherein:

said process water has a pH of about 3.5 to about 5.5.

9. The process of claim 1 wherein:

said wet mat has a moisture content of less than about 70 wt.%.

10. The process of claim 1 wherein:

said dried mat has a moisture content of up to about 10 wt.%.

11. The process of claim 1 wherein:

said binder further comprises a drying oil.

12. The process of claim 1, further comprising:

adding a phenolic resin to said slurry prior to step (b).

13. The process of claim 1 wherein:

said binder consists essentially of an organic isocyanate.

14. The process of claim 1 wherein:

said resin is selected from the group of toluene-2,4-diisocyanate, toluene-2,6-diisocyanate, triphenylmethane triisocyanate, and dimethyl diphenylmethane-4,4-diisocyanate.

15. The process of claim 1 wherein:

said cellulosic filler comprises wood fiber.

16. The process of claim 15 wherein:

said product of step (a) has a moisture content of about 99 wt.%.

17. A process for the preparation of a wood composite, comprising:

(a) forming a slurry comprising water, a cellulosic filler, and a resin comprising 4,4-diphenylmethane-diisocyanate, said slurry having a moisture content of at least about 90 wt.%;

(b) draining sufficient water from the product of step (a) to form a wet mat having a moisture content of less than about 70 wt.%;

(c) removing water from said wet mat by drying to form a dried mat having a moisture content of less than about 10 wt.%; and

(d) pressing said dried mat at a temperature greater than about 400°F to form a molded wood composite.

18. A method for resisting pre-cure in a process for the preparation of a wood composite by a wet-dry forming process wherein a cellulosic mat comprising a binder is formed, comprising:

selecting a binder comprising an organic isocyanate resin.

19. A method for improving the surface quality of a molded wood composite prepared by a wet-dry forming process wherein a cellulosic mat comprising a binder is formed, comprising:

selecting a binder comprising an organic isocyanate resin for use in said wet-dry forming process.

20. A process for the preparation of a wood composite, comprising:

(a) forming a slurry comprising water, a cellulosic filler, and a binder comprising an organic isocyanate resin;

(b) forming a mat from the product of step (a); and

(c) pressing said mat under heat to form a wood composite.

21. The process of claim 20 wherein:

said organic isocyanate resin comprises 4,4-diphenylmethane-diisocyanate.

22. The process of claim 20 wherein:

said mat is pressed at a temperature of at least about 400°F.

23. The process of claim 20 wherein:

said mat is pressed at a temperature of about 430°F to about 500°F.

24. The process of claim 20 wherein:

the weight of said organic isocyanate resin is about 0.25% to about 2% of the weight of said cellulosic filler.

25. The process of claim 20 wherein:

said slurry is formed at a temperature of about 200°F or less.

26. The process of claim 20 wherein:
said mat has a moisture content of at least about 90 wt.%.

27. The process of claim 20 wherein:
said cellulosic filler comprises wood fiber.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US94/09062

A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) :B29C 43/00
US CL :264/109, 320

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 156/62.2, 62.4; 264/109, 320

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	DE,A, 24 44 002 (LEHNERT ET AL.) 25 March 1976, the entire document	2, 12, 14
Y	US,A, 4,407,771 (BETZNER ET AL.) 04 October 1983, column 2, lines 54-62	2, 12, 14
Y	US,A, 4,882,112 (MAKI ET AL.) 21 November 1989, column 2, line 49 to column 3, line 4	1-27
A	US,A, 4,883,546 (KUNNEMEYER) 28 November 1989	

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
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